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210 Timber Jump Lane
Media, PA 19063

April 7, 2004

Steve Alvo
Primary Examiner
Art Unit 1731
Commissioner for Patents
P.O. Box 1450
Arlington, VA 22313-1450

Re. Patent Application No. 10/072,219

Dear Mr. Alvo,

Please find enclosed the above referenced patent application, modified according to the instructions contained in the Office Action Summary mailed to me 03/08/04.

I trust this application is now satisfactory.

Sincerely,

E. Kendall Pye

E. Kendall Pye, Ph.D.

UNITED ST.

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Edward Kendall Pye
210 Timber Jump Lane
Media, PA 19063-1134

EXAMINER	
ALVO, MARC S	
ART UNIT	**PAPER NUMBER**
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Please find below and/or attached an Office communication concerning this application or proceeding.

APPLICATION DATA SHEET

APPLICATION INFORMATION

Application Type::	Regular
Subject Matter::	Utility
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Suggested Group Art Unit::	
CD-ROM or CD?::	None
Title::	Integrated Processing of Biomass and Liquid Effluents
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Request for Early Publication::	No
Request for Non-Publication::	No
Suggested Drawing Figure::	
Total Drawing Sheets::	1
Small Entity::	Yes

APPLICANT INFORMATION

Applicant Authority Type::	Inventor
Primary Citizenship Country::	US
Status::	Full capacity
Given Name::	Edward
Middle Name::	Kendall
Family Name::	Pye

City of Residence:: Media

State or Province of Residence:: PA

Country of Residence:: US

Street of Mailing Address:: 210 Timber Jump Lane

City of Mailing Address:: Media

State or Province of mailing address:: PA

Postal or Zip Code of mailing address:: 19063-1134

Correspondence Information

Name:: Edward Kendall Pye

Street of mailing address:: 210 Timber Jump Lane

City of mailing address:: Media

State or Province of mailing address:: PA

Postal or Zip Code of mailing address:: 19063-1134

Telephone:: (610) 566-6295

Fax:: (610) 566-6295

Domestic Priority Information

Application::	Continuity Type::	Parent Application::	Parent Filing date::
60/307,712	Non-Provisional of	60/307,712	07/26/2001

Foreign Priority Information

Country::	Application number::	Filing Date::	Priority claimed::
Australia	51888/00	08/08/2000	Yes

TITLE OF THE INVENTION,

Integrated Processing of Biomass and Liquid Effluents

CROSS-REFERENCE TO RELATED APPLICATIONS,

Australian Patent Application No: 51888/00; Filing Date: August 8, 2000; Relationship: Equivalent Claims

STATEMENT REGARDING FEDERALLY SPONSORED RESEARCHOR DEVELOPMENT,

Not Applicable

INCORPORATION-BY-REFERENCE OF MATERIAL SUBMITTED ON A COMPACT DISC,

Not applicable

BACKGROUND OF THE INVENTION

This invention relates to a process for the manufacture of multiple valuable products from various wastes generated during the production and recovery of cane sugar, as well from other agricultural cellulosic biomass materials.

The cane sugar industry generates vast quantities of liquid wastes and solid residues during the production of its primary product. These include sugar cane bagasse and liquor streams containing large quantities of low-grade sugars. Bagasse is usually burned inefficiently in boilers to generate steam and power. The liquid waste streams, mostly molasses, are usually sold as low-grade cattle feed additives.

The cane sugar industry is normally a highly-competitive, low profit industry. One approach to improve profitability is to convert these wastes and residues into saleable products that can add to the revenues of the industry. The value of this approach has been recognized for a number of years and considerable research and commercial development has been undertaken to identify useful products, such as furfural and papermaking pulp that can be generated from these wastes.

A new type of pulping technology, known as organosolv pulping, has distinct advantages for the cane sugar industry. It has almost no environmental problems, is

less capital intensive than kraft, it produces multiple co-products, is ideally suited to pulping non-wood biomass that contain high levels of inorganic materials, and can be profitably operated on a much smaller scale than conventional pulping processes. Organosolv pulping has been described in numerous patents and publications including *US Patent No.3,585,104 Kleinert, US Patent No. 4,100,016 Diebold et al., US Patent No. 4,496,426 Baumeister et al.* Although organosolv pulping has numerous advantages over conventional chemical pulping methods, such as the kraft, sulfite and the soda processes, in the pulping of wood and other woody biomass resources, it suffers from a disadvantage of significant losses of the relatively costly organic solvent from the process. If the solvent employed is ethanol the environmental consequences of this loss is minor, but the economic consequences can be important, since they can make the operating costs of an organosolv process higher than those for conventional chemical pulping processes. This requires the provision of substantial quantities of expensive

make-up solvent to the process. This higher operating cost is one major reason that has held back the commercial acceptance of organosolv pulping processes.

One approach to reducing the cost of make-up solvent required by an organosolv pulp mill, to make it economically attractive, is to produce the required make-up solvent on site. For an ethanol-based organosolv pulp mill a fermentation facility can be constructed adjacent to the pulp mill for this purpose. However, if constructed on a small size compatible with the make-up needs of a small pulp mill, the relative capital costs of such a fermentation facility would make it economically unattractive. Furthermore, if the pulp mill uses wood as a raw material the fermentation plant would need to purchase fermentation feedstock, such as sugar or starch, at considerable cost. This problem can be overcome for an ethanol-based organosolv pulp mill processing agricultural residues, such as bagasse. Such a pulp mill processing bagasse would ideally be situated

adjacent to a cane sugar mill, since the bagasse is a low value product of these mills. Therefore no transportation costs would be incurred for the raw material. Another low value by-product of the sugar mill is molasses, a high sugar content liquid by-product. It is well recognized that molasses can be readily fermented commercially to yield ethanol. It therefore represents a low cost fermentation feedstock present at the sugar mill.

Even more attractive for the cane sugar industry is that ethanol-based organosolv pulping is highly compatible with an agricultural economy. Its primary process chemical is ethanol, which can be easily produced by fermentation of waste sugars and starch. Furthermore, many of the co-products of the process find immediate use and value in agriculture, such as animal feed supplements and slow release fertilizer and pesticides. This invention solves the problem of the high capital cost of a dedicated small fermentation plant to provide make-up ethanol by physically

integrating the fermentation of molasses into the process equipment of an organosolv pulp mill.a single mill of organosolv pulping of biomass residues with the processing and fermentation of aqueous waste streams will result in major profits for the cane sugar industry. At the same time such a strategy will result in a high degree of environmental protection and support for the development of adjacent industrial activities based on the co-products of this process.

BRIEF SUMMARY OF THE INVENTION

The present invention provides a process for delignifying biomass fibrous residues comprising:

digesting biomass fibrous residues in a mixture of ethanol and water in a digester at elevated temperature and pressure,

contacting the biomass residues with the mixture of ethanol and water by rotating the spherical digester or by circulating the mixture of ethanol and water between the digester and a holding tank for a circulation time

sufficient to at least partially delignify the biomass fibrous residues and form a pulp,

draining part of the mixture to a spent liquor tank at the end of the circulation time,

causing the remaining liquor and partially delignified biomass to transfer to a blow tank by opening the valve between the digester and the blow tank

The process may include the step of treating spent liquor form the spent liquor tank to recover lignin, acetic acid, furfural, xylose and other co-products, as well as recovering alcohol for re-use in the process and subjecting various liquid waste streams of the process and from a cane sugar mill to fermentation in order to produce ethanol and other fermentation products which may be used in the process.

This process concept is applicable to other biomass residues as well as cane-sugar residues. The potential advantages of integrating these two formerly separate activities into a single operating unit are numerous.

They include lower total capital costs, combining different liquor streams for common product recovery, opportunities for process heat and energy reduction, internal process chemical production, reduced transportation costs, substantial environmental improvement and the potential for the use of larger, more efficient equipment in a shared operation. Separate facilities operating in isolation may not be economic. This identifies the unit processes and the product flows in an integrated total process that would provide these advantages to the cane sugar industry.

Fresh, wet bagasse is brought to the processing plant in a conventional manner. In a preferred embodiment, the water saturated bagasse is transferred to a batch digester and pulped with aqueous ethanol at an appropriate concentration (35%-65% w/w) for about 1-2 hr, at 175°-185°C. This cooking can be done either in one or two stages depending on the type of pulp and properties required. Following cooking, part of the liquor is pumped from the digester to a spent liquor tank and then to a flash

tank from which the alcohol-rich vapours are condensed and recovered.

Following blowing of the pulp and remaining liquor from the digester, the brown-stock bagasse pulp may be passed through a refiner or hydropulper, screened and cleaned by conventional pulp processing equipment and sent on to a bleach plant of appropriate design for the type of pulp desired as the final product. It should be noted here that ethanol-based organosolv pulps are normally more easily bleached than kraft pulps and therefore the bleach plant could be of relatively simple design. Furthermore, organosolv pulps generally have a yield advantage over kraft pulps. The combined effluent from the bleaching stages can then be treated by wet oxidation to produce sodium acetate for sale as a commodity chemical. Because of the integrated nature of the process, wet oxidation of other high BOD concentrated effluents derived from the sugar refining operations can be carried out simultaneously or sequentially in the same equipment. This

would avoid the installation of large-scale effluent treatment systems and furthermore convert a costly treatment step into a revenue source.

The spent aqueous ethanol liquors recovered from the digester are concentrated in the flash tank and combined with acidified stillage from the distillation tower. Acetic acid obtained from a later stage could be used for this acidification. Upon dilution with the acidified stillage, bagasse lignin precipitates from the spent liquor and can be recovered on a drum filter. The lignin cake can be washed on the drum filter with a shower of clean condensate obtained from the stillage evaporator, and then dried in a drier of appropriate design, before bagging and sale. This pure form of lignin has multiple high value uses including as an animal feed efficiency enhancer and as a natural antioxidant in rubber, oils and greases.

The liquor stream from the drum filter can be fed to a distillation column for recovery of ethanol for recycle in the process. Because of normal process losses

there is a need for make-up ethanol. In this invention the required make-up ethanol may be provided internally by first concentrating the distillation tower bottoms (stillage) in an evaporator. After decanting residual lignin and supplementing this liquor with nitrogen and phosphorus-rich effluent from the processing plant, together with waste sugar or starch, it can be fermented by either yeast or anaerobic bacteria to produce ethanol for process make-up or mixed solvents (butanol/acetone/ethanol). Xylose could be recovered from the concentrated stillage prior to this step. In the case of ethanol production the fermented broth can be filtered and combined with the distillation feed stream from the lignin filter and ethanol co-recovered in the same tower. The stillage from this recovery would be rich in acetic acid. Upon evaporation of this stillage an acetic acid-rich condensate can be recovered which can be used partly for lignin washing and the remainder sent to acetic acid recovery. The output from this plant can be used partly to acidify the stillage for lignin precipitation and the remainder

can be sold as food-grade acetic acid, or combined with the wet oxidation liquor for sale as sodium acetate. Market conditions would determine the highest value use for this material. The solids separated from the fermented broth are rich in protein and minerals and could be a suitable animal feed supplement or be used as a fertiliser on the cane fields.

The distillation tower could recover not only ethanol for recycle, but also furfural and ethyl acetate, two valuable products that are generated during the bagasse cooking stage. Both can be sold, probably as crude products suitable for upgrading at a centralised facility. Such activities would encourage the formation of additional local industries designed to support the sugar cane processors using the technology described in this invention. Other local industries could take the lignin produced in these mills and convert it to value-added products, such as concrete admixtures and dye dispersants. Other options presented by this invention include the recovery of xylose (a sugar present in large quantities, mostly as xylan, in bagasse). This could be sold

in the world market for pure xylose that is used as a starting material in the production of the anti-caries sweetener, xylitol. Xylose can also be converted to furfural. If market prices support this option then xylose recovery could be maximised by extended steaming of the bagasse prior to cooking. Xylose would be recovered from the steaming condensate.

Utilising this option would lead to higher value pulps because they would now have high alpha-cellulose content and therefore be suitable for rayon production. The result for the cane sugarl industry of practising this invention would be the elimination of costly environmental control operations and the production of significant revenues from the sale of several value-added products. These products would in turn create opportunities for the introduction of ancillary industries.

BRIEF DESCRIPTION OF THE SEVERAL VIEWS OF THE DRAWING

In the accompanying drawing, which illustrates by way of example the embodiment of this invention, Fig.1 is a flow diagram identifying the unit process steps of an integrated process for the production of pulp and several useful by-products from bagasse, a biomass residue of the cane sugar industry. While not every unit process is essential for the economic success of the invention, the combination of all these unit processes provides maximum utility of the invention.

DETAILED DESCRIPTION OF THE INVENTION.

The present invention is directed at a single integrated process that converts biomass residues from the cane sugar industry, sometimes referred to as bagasse, into a series of valuable products including, but not limited to, papermaking pulps. The process is integrated with the element of fermentation of waste and low grade molasses to produce ethanol, other fermentation products and high protein animal feed. Other sources of low grade, but fermentable carbohydrate may be substituted for

molasses in this invention. A key element incorporated into the process is organosolv delignification of bagasse. This element utilizes some of the alcohol generated in the fermentation element. The organosolv element generates products such as lignin, xylose, furfural, acetic acid and pulp for use in papermaking, dietary fibre, or as chemical cellulose. Much utility is gained by integrating these several elements into a single process. The advantages include heat and energy reduction through process integration, capital reduction through the co-processing of various process streams and waste minimization opportunities through the combining of several process streams. These elements and advantages are illustrated in the process flow diagram, Fig.1.

The process starts with the preparation of the bagasse into a form suitable for packing into a pressure vessel, identified in Fig.1 as a digester. The preferred form is into stem sections of approximately ten centimeters in length, but any similar form is appropriate. A compressed pellet is also appropriate as feed for the digester. As illustrated the digester is

one of a series of batch digesters that may be rotating spheres, or a continuous conveyed inclined or horizontal tube configuration, but could also be a vertical tubular batch or continuous design. In Fig.1 a preferred configuration of a rotating globe batch digester configuration is illustrated. The operations described below for one digester are identical to those for the additional digesters that are operated sequentially at appropriate intervals to allow optimal use of the remaining equipment in the process.

Following preparation of the fibrous residue (bagasse) into the useful form described above it is conveyed to the top of the digester for loading into the digester. Once the digester is filled with a pre-determined amount of bagasse, the conveyer is stopped and the digester is closed. An exhaust valve located behind a screen in the bottom of the digester is now opened and low-pressure steam (less than 50 psi) is allowed to enter the top of the digester. This steaming, which is required to displace air in the fibre bed, continues until temperature sensors in the exhaust line indicate that steam is exiting from the bottom of

the digester. Any condensate of the steaming exits through the same line. Alternatively, if the moisture content of the bagasse is too high, nitrogen gas may be substituted for steam for the air displacement. All valves are now closed and the pump in the line exiting the 2nd liquor tank is turned on. The 2nd liquor tank is full of aqueous alcohol at the desired concentration and temperature. This liquor was used as a wash liquor from a previous digester cook and was retained between cooks in the 2nd Liquor Tank. The preferred alcohol concentration in water is in the range of 35%-70% (w/w) and the preferred temperature is in the range of 170°-205°C. This liquor is pumped through a heat exchanger to maintain its desired temperature and then into the top of the digester through the top liquor line. Once the digester starts to fill, liquor exits the bottom of the digester from behind a screen constructed around the outlet line at the bottom of the digester, from where it is returned to the top of the 2nd Liquor Tank. This hot liquor circulation is continued for the appropriate time necessary to raise the contents of the digester to the desired cooking temperature. At this point the liquor exit

valve is closed and the desired weight of hot liquor, usually 2 to 5 times the dry weight of the bagasse, is pumped into the digester from the 2nd Liquor Tank. Liquor flow is then stopped, steam is continually sent to a jacket surrounding the digester to maintain its temperature at the desired cooking temperature and the globe digester is rotated for the desired cooking time. This time is normally between 30 minutes and 3 hours, with the preferred time being between one and two hours. At the end of this time the rotation of the digester is stopped with the liquor outlet line and surrounding screen at the bottom of the digester. Part of the hot black liquor is then flashed into a Flash Tank. The valve at the top of the 2nd Liquor Tank is then closed and the return liquor is diverted to the Spent Liquor Tank. Residual liquor in the 2nd Liquor Tank is pumped down to a level sufficient to keep the suction side of the pump flooded. Liquor remaining in the Digester is drained through the lower screens into a drain line from where it is also pumped to the Spent Liquor Tank. Next, clean aqueous alcohol at the concentration and temperature previously described is pumped from the 1st Liquor

Tank into the top and bottom lines of the Digester and returned to the 2nd Liquor Tank using the appropriate valves and pumps. After the 1st Liquor Tank has been almost emptied liquor flow to the Digester is stopped and the remaining liquor in the Digester is drained down and pumped to the 2nd Liquor Tank through the appropriate lines and valves. The Digester is now depressurized by opening the valves in the top line and the vapors passed to a Blow-Down Condenser. The alcohol-rich vapors are condensed and returned to the Recovery Alcohol Tank for re-use in the process. The partially-delignified fibres are now sluiced from the Digester through the bottom valve, using water or preferably condensate from the evaporator. This sluiced pulp is sent to a tank from which it is pumped continuously to conventional pulp refining, washing, screening, cleaning and bleaching operations. The liquor from these operations can be processed by conventional means for alcohol recovery and sodium acetate recovery.

The spent liquor under pressure in the Spent Liquor Tank is flashed into a Flash Tank and the vapors condensed

through the Blow-Down Condenser and returned to the Recovery Alcohol Tank for re-use. The condensed liquor in the Flash Tank, containing the extracted lignin, is then pumped to the Lignin Precipitation Tank where it is mixed rapidly with stillage from the Distillation Tower and the pH adjusted to below 3.0 with acid and the mixture cooled to about 17°. Lignin precipitates from the mixture and forms a slurry. This is pumped to a suitable filtering device, such as a drum filter, where the lignin is removed as a wet cake that is sent to an appropriate drier, while the filtrate is pumped to a Recovery Feed Tank. From this tank the filtrate is pumped to an appropriately designed Distillation Tower. Such a tower would have a lower steam stripping section and an upper rectifying section, or be composed of two columns having these functions. In this tower alcohol, together with some esters, is recovered as an overhead condensate and returned to the Recovery Alcohol Tank for re-use in the process. Furfural, which is present in the filtrate, accumulates at one of the lower trays in the rectifier section where it is drawn off, cooled and mixed with water

before being sent to the Decanter. The lower liquid phase in the decanter is crude furfural, which is up-graded to merchant furfural in a commercially available system. The upper layer is aqueous alcohol, which is passed back to the Distillation Tower to recover the alcohol by mixing with the tower feed stream. Steam to power the stream stripping section is provided by Reboilers at the bottom of the tower.

Aqueous stillage from the bottom of the Distillation Tower containing sugars, some lignin and minerals, is sent to an appropriately designed multi-stage evaporator where it is concentrated to about 25% solids. This concentrate is pumped to an Evaporator Concentrate Decanter where a lower layer of oily lignin is recovered and dried. The upper aqueous layer containing xylose, xylans, other sugars and minerals, is sent to a commercially available xylose recovery unit for production of purified xylose. The effluents from this unit include waste hexose sugars, which are passed to the fermentation operations for alcohol production, and an aqueous solution of minerals, which are returned to the cane fields as fertilizer.

The aqueous condensate from the Evaporator is passed to a commercially-available solvent extraction unit, such as those employing tri-octyl phosphine oxide, (TOPO), for recovery of acetic acid, formic acid, furfural and ethanol as separate marketable products. The clean water that exits this unit is useful in the pulp washing and bleaching operations.

Molasses is used as the fermentation raw material in a fermentation plant employing yeast to produce ethanol and other fermentation products. This medium may be supplemented with additional sugars from acid-hydrolysis products of waste cellulose, as required for maximum productivity. Additional minerals that may be required at this stage can be supplied from the waste liquor stream of the xylose recovery plant. After fermentation is complete in a sequential battery of batch Fermenters, the beer is pumped to a Filter where yeast and other solids are removed and dried for sale as high-protein animal feed supplement. The clarified beer is then passed to the Recovery Feed Tank, mixed with the filtrate from

the Lignin Recovery Filter and pumped to the Distillation Tower for recovery of ethanol and other components.

By this invention a range of valuable products is produced from the solid and liquid residues of the cane sugar industry.

CLAIMS.

1. An integrated process performing fermentation and the conversion of biomass residues (bagasse) from the cane sugar industry for the production of multiple useful products; such process combining the elements of

alcohol-based organosolv pulping and

ethanol fermentation

into a single integrated process.

2. A process, as in claim 1, in which the useful products are any combination, or number, of products including

unbleached papermaking pulp,

bleached papermaking pulp,

high alpha-cellulose pulp,

organosolv lignin, furfural,

acetic acid,

ethyl acetate,

sodium acetate,

xylose,

xylan,

butanol,

acetone,

high-protein animal feed,

plant fertilizer,

uronic acids and

ethanol.

ABSTRACT OF THE DISCLOSURE.

An integrated process for the combined fermentation and the conversion of liquid and solid residues generated by the cane sugar industry into a variety of useful products. The process also relates to the treatment of other biomass materials. The process combines elements of alcohol-based organosolv pulping with fermentation in a fully integrated operation. This process eliminates the need for separate costly facilities, thus reducing capital and operating costs, providing opportunities for heat and energy reduction and a high degree of internal process recycle. The process would replace costly waste and residue treatment operations with revenue generating operations.

SEQUENCE LISTING

Not Applicable